IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
Docket No. 12329US03

In the Application of:

ROGER BERNARDS, HECTOR GONZALEZ, AL KUCERA and MIKE SCHANHAAR

U.S. Serial No.: 10/028,955

Filed: December 18, 2001

For: METHOD FOR ROUGHENING COPPER SURFACES FOR BONDING TO SUBSTRATES

Examiner: S. Ahmed

Group Art Unit: 1765

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail, postage prepaid, in an envelope addressed to: Commissioner for Patents, Washington, D.C. 20231, on this 15th day of November, 2004.

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DECLARATION UNDER 37 C.F.R. § 1.131

Commissioner for Patents
P.O. Box 1450
Alexandria Virginia 22313-1450

Sir:

We, Roger Bernards, Hector Gonzalez, Al Kucera, and Mike Schanhaar, declare the following:

1. We are the applicants of the above-identified patent application and coinventors of the subject matter described and claimed therein.

2. Prior to the filing date of United States Patent No. 6,666,987 ("Morikawa"), which is June 8, 1999, we reduced to practice the idea of a process for preparing roughened copper surfaces comprising the steps of contacting with a clean copper surface an adhesion promoting composition under conditions effective to provide a

roughened copper surface, wherein the adhesion promoting composition consists essentially of hydrogen peroxide, a pH adjuster, a topography modifier, and a uniformity enhancer, and at least essentially free of halogen ions.

3. Prior to the filing date of Morikawa, we completed our invention as described and claimed in at least claim 1 of the present application, as evidenced by the laboratory notebook pages attached as Exhibit A.

4. The laboratory notebook pages are those of one of the undersigned applicants, Roger Bernards.

5. The first page (laboratory notebook page 29) describes the commencement of a multi-day experiment designed to develop a process for preparing roughened copper surfaces.

6. As shown on this page, a standard bath was used for each set of runs in the experiment. The standard bath contained:

(a) H_2O_2 (hydrogen peroxide),

(b) H_2SO_4 (sulfuric acid), and

(c) $CuSO_4$ (copper sulfate).

7. The run "sets" that follow on the first and subsequent pages describe additional components that were added to the standard bath prior to applying the composition to a copper surface.

8. Set "K" is described on page 34. The heading for Set K indicates the component that was added to the standard bath for this set of runs: "5 Amino Tetrazole • H_2O".

9. Set K consists of 13 runs. Each run lists the concentration of 5-aminotetrazole added to the standard bath, plus any additional components that were added. Run 4 lists: ".5 g/L + 1.5 g/L BTA". BTA is an acronym for benzotriazole. In run 4, therefore, 0.5 g/L of 5-aminotetrazole and 1.5 g/L of benzotriazole were added to the standard bath.

10. Thus, the composition used in Run 4 of Set K contained the following components:

(a) hydrogen peroxide,

(b) a pH adjuster (sulfuric acid),

(c) a topography modifier (benzotriazole),

(d) a uniformity enhancer (5-aminotetrazole), and

(e) copper sulfate.

11. As explained on laboratory notebook page 34, the composition of Run 4 was successful when applied to a copper surface: "Dark and very uniform looks great" and "What's awesome about #4 and #9 and others on this page is that no matter how [expletive] the surface is before going into the etch you get complete coverage with no skip etch."

12. Each of the dates deleted from Exhibit A is prior to the filing date of Morikawa.

13. We certify that all statements made herein of our own knowledge are true, and that all statements made herein on information and belief are believed to be true. We understand that willful false statements and the like are punishable by fine or

imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.

Roger Bernards — Date: 10/5/04

Hector Gonzalez — Date: 10/25/04

Al Kucera — Date: 10/8/04

Mike Schanhaar — Date: 10-5-04

Objective To make a dark coating with Cobra bond + additives other Than BTA

Standard Bath: H_2O_2 : 3% Temp 98°F (from 5% H_2SO_4 add) 94°F-97°F
H_2SO_4 : 5%
$CuSO_4 \cdot 5H_2O$: 40 g/L
Cl^- : Zero unless listed
Additive : Variable

3-X Set A : Cobratech 928 — Material is only 90% active (not taken into account for g/L)

		Gross	Tare	Dwell (min)	Etch	Appearance
1)	1 g/L	1.5870	1.5159	1:10	80.5	Slightly darkened
2)	3 g/L	1.5916	1.5326	1	67	Lighter but cool looking
3)	5 g/L	1.5671	1.5165	1	57.	same
4)	7 g/L	1.5607	1.5148	1	52	same ...
5)	10 g/L	1.5372	1.4891	1	54	Same
6)	15 g/L	1.6355	1.5866	1	55	" maybe not as rough
7)	25 g/L	1.6315	1.5857	1	52	" maybe not as rough
8)	0.5 g/L	1.5764	1.5172	1	67	not as rough Looking

3-X SET B Cobratec PT (The PT Solution has 10% H_2SO_4)

1)	~~1g/L~~ 0 g/L	1.5997	1.5328	1	76	looks very smooth
2)	1 g/L (active Material)	1.5956	1.5520	1	49.	Striations on top side Bottom side looks not etched
3)	2 g/L					
4)	2 g/L Repeat	1.6062	1.5338	1:20	82	Striations
5)	2 g/L + 5 g/L Cl^-	1.6068	1.5995	1:20	8.3	No etch
6)	3 g/L	1.5963	1.5225	1:20	84	Striations Big time
7)	4 g/L	?	1.5388	1:28 x 80		Striations
8)	4 g/L	1.5921	1.5288	1:20	72	"
9)	5 g/L	1.6065	1.5355	1:20	80	"
10)	7 g/L	1.5990	1.4924	1:20	121	"
10)	10 g/L	1.5701	1.5211	1:00	55	"
11)	15 g/L adjusted for H_2SO_4	1.5791	1.4745	1:50	118	"
12)	1.5 g/L	1.6021	1.5227	1:20	90	non uniform thin marker
13)	2.5	1.5961	1.5071	1:20	101	Striations Fat marker

Cont. Pg 30

WITNESSED AND UNDERSTOOD
SIGNED Lee Burger [redacted] RB
SIGNED [redacted]

From pg 29

10.61

3X Set C — TT100 (Cobratec TT100)

	Gross	Tare	Total Etch	Dwell	Appearance
1) 0 g/L	1.5813	1.4838	110	1:25	dull copper
2) ~~1~~ .36/L	1.5981	1.5061	104	1:20	
3) 1 g/L	1.5982	1.4978	114	1:20	
4) 2 g/L	1.5998	1.5112	91	1:20	
5) 3 g/L	1.5958	1.5083	99	1:20	
6) 4 g/L	1.5515	1.4560	108	1:23	
7) 6 g/L	1.5454	1.4644	92	1:20	
8) 10 g/L	1.5770				

6X Set D — Alpha prep PC ~~7042~~ 7042

1) 50% + 3% H_2O_2	1.5907	1.5660	28	1:20	look oxidized
2) " + 6% H_2O_2	1.5787	1.5352	49	1:20	striations
3) " + 9% H_2O_2	1.6033	1.5281	85	1:20	BTA on surface
4) " + 9% H_2O_2 + 4.3.9% H_2SO_4 (5% total)	1.5909	1.5075	100	1:20	striations
5) " + 9% H_2O_2 + 3.9% H_2SO_4 + 13.5 g/L $CuSO_4 \cdot 5H_2O$	1.5770	1.4993	88	1:30	Bath turned green

6X SET E — NaBr at 8.5 g/L BTA

1) 4 g/L 3% H_2O_2	1.5857	1.5776		1:20	
2) " 6% H_2O_2	1.5956	1.5843	13	1:20	
3) " 9% H_2O_2	1.6040	1.5852	21	1:20	~~Looks like cobra with Cl~~
4) " + 12% H_2O	1.5867	1.4727	129	1:20	Looks Like Cobra w. Cl^- Stained
5) 12 g/L ~~H~~ 14% H_2O_2	~~1.5826~~ ~~1.5935~~ 1.6149	1.5348	91	1:20	" "

Cont. on pg 32

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Lee Bugler [redacted] RB [redacted]

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 PROJECT From pg 30 PROJECT NO.

6) 0.53g/L NaBr 1.5739 1.5138 68 1:20 Stained like C1
+ 14% H_2O_2
+ 8.53g/L BTA

~~6 Glicoat F-1 1.5879 1.5878~~
~~+ 5% H_2SO_4~~
~~+ 3% H_2O_2~~

SET F Glicoat F-1 Lot 961205 exp [redacted]

Normal conditions ie 5% H_2SO_4 + 3% H_2O_2 + 40g/L $CuSO_4 \cdot 5H_2O$, 98°F
+

25% by vol F-1	1.5872	1.5866	0.7	1:30	Bright water beads up on it
25% + 9% H_2O_2	1.6080	1.6076	still no etch		"
10% + 3% H_2O_2	1.6049	1.6040			
10% + 14% H_2O_2	1.6191	1.6140	6	2:00	Still no etch
2% + 3% H_2O_2	1.5761	1.5698	7	1:20	
2% + 14% H_2O_2	1.5752	1.5443	35	1:20	Still Bright but not as shiney

Part gasses ~~like crazy~~
but still Hardly no etch
H_2O_2 Breaking down by
Side Rx

6X → SET G Glicoat E2L

2% + 3% H_2O_2	1.5728	1.5446	32	1:20	Bright but not shiney
2% + 6% H_2O_2	1.6025	1.5034	112	2:00	Bright but not shiney

Part really gassing ~~no~~ not much etch

Cont. Pg 33

WITNESSED AND UNDERSTOOD
SIGNED Lee Burger DATE [redacted] SIGNED RB [redacted]
SIGNED DATE DATE

2X SET H 1- Methyl imidizole normal conditions unless otherwise stated

1)	5g/L	1.5998	1.5803	22	1:20	Bright Shiney
2)	5g/L + 8.5 BTA	1.5797	1.5150	73	1:20	not as Dark as just BTA
3)	.5g/L + ~~8.5 BTA~~ + 3.0 BTA sh		1.5056			fairly Dark

3X SET I 2,1,3 Benzothiadiazole

1)	5g/L	1.6117	1.5537	66	1:30	Light
	Not all dissolved looks completely insoluble					
2)	5g/L + 3g/L BTA	1.5755	1.5043	81	1:20	Slightly Dark

3X SET J 1H-[1,2,3 Triazolo(4,5-b] pyridine

1)	5g/L	1.5982	1.5435	62	1:20	Big time striations Looks Like fire
2)	5g/L + 3g/L BTA	1.5900	1.5296	68	1:20	Brown
3)	10g/L	1.5983	1.5385	68	1:20	striations Bad
4)	10g/L + 3g/L BTA	~~1.5535~~ ~~1.5383~~			1:20	Still some striations
5)	.5g/L	1.5972	1.5169	91	1:20	Striations
6)	.5g/L + 3g/L BTA	1.5748	1.4955	90	1:25	Looks Like BTA only Marker spreads

Cont. on Pg 34

WITN SIGN Lee Burgh [redacted] RB [redacted]

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SUBJECT from pg 33 PROJECT NO.

2X SET K 5 Amino Tetrazole $\cdot H_2O$.

1) 5g/L	1.5839	1.5313	60	1:20	Light but rough
2) 5g/L + 3g/L BTA	1.5744	1.5096	73	1:35	Reddish Looking Marker spreads
3) 0.5g/L	—			1:20	Looks Like BTA Little Lighter
4) .5g/L + 1.5g/L BTA	1.4935	1.3685	142	1:50	Dark and very uniform looks great
5) .5g/L + 3g/L BTA	~~1.4979~~	~~1.4979~~		1:20	Dark ~~or~~ put not uniform skip
6) 0.25g/L	1.5798				~~Da~~ Lighter fairly uniform
7) .25g/L + .5g/L BTA	1.6148	1.5419	82		Lighter fairly uniform
8) .25g/L + 1g/L BTA	1.5892	1.5212	~~.068~~ 77	1:20	Kinda Dark ~~fairly uniform~~ Not uniform
9) .5g + .5g BTA	1.5777	1.5046	83	1:28	Dark even not as Dark as #4

Note Whats Awesome about #4 and #9 (and others on this page) is that no matter how shitty the surface is before going into the etch you get complete coverage with no skip etch

10) .5g/L + 1g/L BTA	1.6180	1.5552	71	1:20	Dark even, not as Dark as 4
11) 1g/L + 1g/L BTA	1.5845	1.5280	64	1:20	Down side is Darker than up not as Dark as 10
12) 1g/L + 2g/L BTA	1.6051	1.5348	80	1:27	Dark uniform Complete Coverage
13) 1g/L + 3g/L BTA	1.6103	1.5432	76	1:21	

Cont on pg 34

WITNESSED AND UNDERSTOOD

SIGNED Lee Burgell DATE [redacted] SIGNED RB [redacted]

SIGNED DATE DATE

DATE [redacted] SUBJECT Conclusions of Work on pages 27-47 PROJECT NO. 69

The main conclusion drawn from the testing of the many additives from pages 27-47 can be found on pg 34 from ~~Set K~~ (Set K), See the note entered on that page. The 5 Amino tetrazole acts as a uniformity enhancer if it is in the right concentration and if it is in the right ratio with The BTA. This is a big improvement over the BTA only system and may be patentable. Further testing by others at electrochemicals [See Al Kucera's, Hector gonzalas' and Mike schanhaar's Notebooks] Also show that the 5 amine tetrazole acts as a unifermity enhancer. ~~&~~ ie the panels look more uniform in color, degree of etch, and coverage of the coating when the 5 amino tetrazole is used in conjuction with the BTA. Also the 5 amino tetrazole when used as the sole additive is not as uniform as when used in conjuction with the BTA. On pg-34 #4 is definately better than #1 as ~~wb~~ was noted when I ~~said~~ wrote #4 "looks great." The optimum Ratio of BTA to 5amine tetrazole is close to 3 to 1 and the best concentration of BTA is close to 1.5 g/L and the best concentration for 5 amino tetrazole is closer to 0.5 g/L The H_2O_2, H_2SO_4, Temp., Copper (copper can be zero, copper is not needed to make this formulation work properly.) ~~Concentrations~~ concentrations can vary a lot but I think H_2O_2 = .2% - 5%, H_2SO_4 .1% - 8%, and Temp 60°F - 130°F is the best range. Most commonly H_2O_2 = 1% - 2%, H_2SO_4 4 - 6%, and Temp 80 - 100°F is employed.

Roger Bacon

WITNESSED AND UNDERSTOOD BY [redacted]

Lee Bright

RB [redacted]